

The use of a dispersion stabilizer in the preparation of crosslinked polymer particles is essential for preventing particle coagulation during polymerization. Various synthetic high molecular weight compounds including homopolymers, copolymers, graft polymers, block copolymers, and the like, can be used as a dispersion stabilizer. In addition to poly(vinyl pyrrolidone) other suitable dispersion stabilizers are polyisobutylene succinimide, poly(methyl-methacrylate)-g-poly(12-hydroxy stearic acid) and the like.

The monomer, styrene, can be substituted by different vinyl monomers such as methyl methacrylate, vinyl acetate, acrylate, ethyl vinylbenzene, vinylpyridine, acrylonitrile, and the like, to produce final particles with different properties such as density, reflective index, heat resistance, solvent resistance, . . .etc. The crosslinker, divinylbenzene, can also be substituted for with other compounds having two or more than two polymerizable double bonds such as nonconjugated divinyl compounds, diacrylate compounds, triacrylate compounds, dimethacrylate compounds, trimethacrylate compounds, and the like.

Other radical polymerization initiators which can be used in the two stage dispersion polymerization are 4,4'-azobis(r-cyanopentanoic acid), 2,2'-azobis(2-methylbutyronitrile), benzoyl peroxide, lauroyl peroxide, octanoyl peroxide, and the like.

The dispersion medium, methanol, can be substituted for with other alcohols such as ethyl alcohol, propyl alcohol, butyl alcohol, amyl alcohol, benzyl alcohol, ether alcohol, and the like, or hydrocarbons such as hexane, octane, decane, cyclohexane, xylene, and the like, or with ethers, halogenated hydrocarbons, ketones, esters and the like.

In addition, the ratio of the monomer and crosslinker in the Table I can be varied to determine the physical properties of the final particles, such as density, reflective index, heat resistance, solvent resistance, particle size and so on.

After the second stage polymerization process, the final polymer

particles are transferred from the dispersion medium to a desired dielectric medium by a washing process which comprises mixing the final product with a solvent which is mixable with both the dispersion medium and the desired dielectric medium, centrifuging the mixture to separate the particles and the liquids and decanting the supernatant to yield the concentrated particles. The washing procedure is repeated several times until the methanol is completely removed. The solvent washed particles are then dispersed in the desired dielectric medium with the addition of a charge control agent and a dark color dye. Sometimes, a stabilizer or a co-stabilizer is added to the final dispersion to help prevent the particles from coagulating. Therefore, the final dispersion contains electrophoretic particles having a positive electrophoretic mobility dispersed in a dark color-dye solution which gives good optical contrast with the white particles, and is ready to be used in EPIDs.

In the case of black particles, the metal oxide stained particles are transferred from the aqueous staining solution to a desired dielectric medium by the same washing process as outlined above. After being transferred to the desired medium, a charge control agent, sometimes together with a stabilizer or a costabilizer, is added to the dispersion to produce a final black dispersion containing positively charged black particles suspended in a clear medium, which is ready to use in EPIDs.

The preferred dielectric media for the final dispersion are non-polar solvents such as tetrachlorethylene, carbon tetrachloride, pentane, octane, decane, cyclohexane, benzene, xylene, Isopars, sec-butylbenzene, and the like, or a mixture of the liquids. The solvents used for washing the particles should be mixable with both the dispersion medium and the dielectric medium, examples are ethanol, propanol, butanol, acetone, tetrahydrofuran, ketones, ethers, esters, and the like. The desirable charge control agents for positive charging are surfactants with an acidic character such as polyisobutylene succinic anhydride, cupric naphthenate, zirconium octoate, zinc octoate, calcium octoate, polyvinyl

alcohol, polyacrylic acid, polymethacrylic acid, polyvinyl chloride, and the like. A dark colored dye such as Solvent Blue, Oil Blue A, Sudan Black B, Sudan Red 7B and the like, is preferred. The preferable stabilizers or co-stabilizer used with the charge control agents are high molecular weight compounds including homopolymers, copolymers, graft polymers, block copolymers, natural high molecular compounds, and the like. Specific examples of suitable stabilizers are poly(12-hydroxystearic acid)-graft-poly(methyl methacrylate-methacrylic acid), polystyrene-co-poly(vinyl pyridine), poly(vinyl alcohol)-co-polyethylene, and the like.

EXAMPLE TWO

In an alternative embodiment of the present invention, white, electrophoretic, highly crosslinked polymer particles 22 with controlled surface functionality, are prepared by a two-stage dispersion polymerization technique. The polymerization recipe is listed in Table II.

Table II

<u>Materials</u>	<u>Weight (g)</u>
Methanol	100
divinylbenzene	5
styrene	5
2,2'-azobisisobutyronitrile	0.5
poly(acrylic acid)	2
methacrylic acid	0.5

As in process one, the inhibitors of the monomer styrene and the crosslinker divinylbenzene are removed by washing with 10% NaOH aqueous solution several times, drying with calcium carbonate over night at 0°C and then passing through a column containing an appropriate inhibitor remover. The inhibitor-free monomers and crosslinker are stored in a freezer for later use. In this, and subsequent processes, the styrene is of the type commercially available from

5 Fisher Scientific, Inc. and the divinylbenzene is of the type commercially produced by Dow Chemical, Inc. The initiator, 2,2'-azobisisobutyronitrile (AIBN) is manufactured by Kodak Co. The second stage monomer, methacrylic acid and the stabilizer, poly(acrylic acid), are of the types commercially available from Aldrich Chemical Co. The dispersion medium, methanol, a reagent grade, is from Fisher Scientific, Inc.

10 The styrene and divinylbenzene are mixed with methanol and charged to a closed container containing the AIBN and poly(acrylic acid) which are carefully weighed. The closed container is purged with nitrogen by bubbling through the solution for a certain time. The container is then warmed and agitated for a desired reaction time. In one preferred embodiment, the mixture is tumbled at thirty revolutions per minute for eight hours at sixty degrees celsius. After eight hours of tumbling, the second stage monomer, methacrylic acid, is injected in the container which continues to tumble at the same reaction condition for another desired reaction time. The final product made by the two stage dispersion polymerization process is highly crosslinked poly(styrene-co-divinylbenzene) particles with poly(methacrylic acid) grafted on the surface. The particle size of the final particles is uniform and varied from 0.1 to 5 μm depending upon the reaction media used. The final particles are dielectric and have a density close to 1 g/cm^3 . The polymer particles are separated from the dispersion medium by centrifuging and decanting the dispersion medium.

20 To form black particles, the particles of the previous process are then mixed and tumbled with a osmium tetroxide aqueous solution at room temperature for a desired reaction time. The osmium tetroxide reacts with and stains the residual double bonds of the poly(styrene-co-divinylbenzene) particles, thereby resulting in highly crosslinked polymer particles having a desired degree of blackness that can be used as the present invention electrophoretic particles 122. It should be understood that in place and stead of the osmium tetroxide, ruthenium tetroxide or other metal oxides may also be used.

By varying the polymerization recipe of Table II and by varying other reaction parameters of the method of manufacture, the physical characteristics of the white and black particles produced can be selectively altered as needed for a given application. The surface functionality of the final particles can be varied by introducing different functional monomers, such as acrylic acid, sodium styrene sulfonate, maleic acid, chlorostyrene, vinyl alcohol, and the like, at the second stage polymerization to produce poly(styrene-co-divinylbenzene) particles with acidic surface characteristics which are suitable for developing negative electrophoretic mobilities in dielectric media.

The use of a dispersion stabilizer in the preparation of crosslinker polymer particles is essential for preventing particle coagulation during polymerization. Various synthetic high molecular weight compounds including homopolymers, copolymers, graft polymers, block copolymers, and the like, can be used as a dispersion stabilizer. In addition to poly(acrylic acid), other suitable dispersion stabilizers are poly(vinyl alcohol), poly(methacrylic acid), sorbitan stearic acid monoester, and the like.

The monomer, styrene, can be substituted for by different vinyl monomers such as methyl methacrylate, vinyl acetate, acrylate, ethyl vinylbenzene, vinylpyridine, acrylonitrile, and the like to produce final particles with different properties such as density, reflective index, heat resistance, solvent resistance, . . . etc. The crosslinker, divinylbenzene, can also be substituted for by other compounds having two or more than two polymerizable double bonds such as nonconjugated divinyl compounds, diacrylate compounds, triacrylate compounds, dimethacrylate compounds, trimethacrylate compounds, and the like.

Other radical polymerization initiators which can be used in the two stage dispersion polymerization are 4,4'-azobis(4-cyanopentonic acid), 2,2'-azobis(2-methylbutyronitrile), benzoyl peroxide, lauroyl peroxide, octanoyl peroxide, and the like.

The dispersion medium, methanol can be substituted for with other

alcohols such as ethyl alcohol, propyl alcohol, butyl alcohol, amyl alcohol, benzyl alcohol, ether alcohol, and the like, or with hydrocarbons such as hexane, octane, decane, cyclohexane, xylene, and the like, or with ethers, halogenated hydrocarbons, ketones, esters and the like.

5 In addition, the ratio of the monomer and crosslinker in the Table II can be varied to determine the physical properties of final particles, such as density, reflective index, heat resistance, solvent resistance, particle size and so on.

10 After the second stage polymerization process, the final polymer particles are transferred from the dispersion medium to a desired dielectric medium by a washing process which comprises mixing the final product with a solvent which is mixable with both the dispersion medium and the desired dielectric medium, centrifuging the mixture to separate the particles and the liquids and decanting the supernatant to yield the concentrated particles. The
15 washing procedure is repeated several times until the methanol is completely removed. The solvent washed particles are then dispersed in the desired dielectric medium with the addition of a charge control agent and a dark color dye. Sometimes, a stabilizer or a co-stabilizer is added to the final dispersion to help prevent the particles from coagulating. Therefore, the final dispersion
20 contains white electrophoretic particles having negative electrophoretic mobility dispersed in a dark color dye fluid which gives a good optical contrast with the white particles, and is ready to be used in EPIDs.

25 In the case of black particles, the metal oxide stained particles are transferred from the aqueous solution to a desired dielectric medium by the same washing process described above. After being transferred to the desired medium, a charge control agent, sometimes together with a stabilizer or a costabilizer, is added to the dispersion to produce a final black dispersion containing negatively charged black particles suspended in a clear medium, which is ready to use in EPIDs.

The preferred dielectric media for both the black and white dispersion are non-polar solvents such as tetrachloroethylene, carbon tetrachloride, pentane, octane, decane, cyclohexane, benzene, xylene, Isopars, sec-butylbenzene, and the like or a mixture of the liquids. The solvents used for washing the particles should be mixable with both the dispersion medium and the dielectric medium, examples are ethanol, propanol, butanol, acetone, tetrahydrofuran, ketones, ethers, esters, and the like. The desirable charge control agents for negative charging are surfactants with a basic character such as polyisobutylene succinimide, barium petronate, barium sulfonate, barium dinonylnaphthalenesulfonate, metal oxides, polyvinyl pyridine, pyridine, lecithin, polyvinyl acetate, polyethylene oxide, polymethy methacrylate, polyacrylamide, polyesters, polyethers, and the like. A dark colored dye such as Solvent Blue, Oil Blue A, Sudan Black B, Sudan Red 7B, and the like is preferred. The preferable stabilizers or co-stabilizer used with the charge control agents are high molecular weight compounds including homopolymers, copolymers, graft polymers, block copolymers, natural high molecular compounds, and the like. Specific examples of suitable stabilizers are poly(12-hydroxystearic acid)-graft-poly(methyl methacrylate-methacrylic acid), polystyrene-co-poly(vinyl pyridine), poly(vinyl alcohol)-co-polyethylene, and the like.

As will be recognized by a person skilled in the art, the highly crosslinked polymer particles having surfaces with controlled acidic moieties or basic moieties, made by emulsion polymerization, miniemulsion polymerization, microemulsion polymerization, suspension polymerization, precipitation, seeded emulsion polymerization or seeded dispersion polymerization, can also be used as white electrophoretic particles, or be used as black electrophoretic particles after being stained with a metal oxide in electrophoretic image displays.

All equivalents, variations and modifications that can be applied to the described present invention by a person skilled in the art, are intended to be included within the scope of this invention as defined by the appended claims.

Claims:

1. A process for forming dielectric particles, comprising:
 - (a) providing a dispersion medium;
 - (b) polymerizing a first monomer in said medium to form
5 polymer particles;
 - (c) polymerizing a second monomer in said medium, said
polymerized second monomer grafting upon said polymer particles; and
 - (d) controlling the surface functionality of said particles.
- 10 2. The process of Claim 1 wherein said medium is a liquid, wherein
said step (b) includes
 - admixing a first monomer and a crosslinker with said liquid
dispersion medium forming a first mixture;
 - preparing a second mixture of an initiator and a stabilizer;
 - 15 adding said first mixture to said second mixture to form a third
mixture; and
 - allowing said first monomer to polymerize within said third
mixture to form said polymer particles, and wherein said step (c) includes
introducing said second monomer to said third mixture, said second
20 monomer at least partially polymerizing and grafting upon said polymer particles.
3. The process of Claim 2, wherein said step of controlling includes

the step of introducing a functional monomer when said second monomer is introduced.

4. The process of Claim 3, wherein said first monomer is a vinyl
5 monomer.

5. The process of Claim 3, wherein said first monomer is selected
from the group consisting of styrene, methyl methacrylate, vinyl acetate, acrylate,
ethyl vinylbenzene, vinylpyridine, and acrylonitrile.

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6. The process of Claim 5, wherein said crosslinker is a compound
having a plurality of polymerizable double bonds.

7. The process of Claim 6, wherein said crosslinker is selected from
15 the group consisting of nonconjugated divinyl compounds, diacrylate compounds,
triacylate compounds, dimethacrylate compounds and trimethacrylate
compounds.

8. The process of Claim 7, wherein said initiator is selected from the
20 group consisting of 2,2'-azobisisobutyronitrile, 4,4'-azobis(r-cyanopentanic acid),
2,2'-azobis(2-methylbutyronitrile), benzoyl peroxide, lauroyl peroxide and
octanoyl peroxide.

9. The process of Claim 8, wherein said stabilizer is selected from the group consisting of poly(vinyl pyrrolidone), homopolymers, copolymers, graft polymers, block polymers, poly(acrylic acid), poly(vinyl alcohol), poly(methacrylic acid) and sorbitan stearic acid monoester.

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10. The process of Claim 9, wherein said second monomer is acrylamide.

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11. The process of Claim 10, wherein said functional monomer is selected from the group consisting of vinyl acetate, methyl methacrylate, acrylonitrile, N-(iso-utoxymethyl) acrylamide and dimethylaminopropylmethacrylamide and produce dielectric particles with a basic surface suitable for positive charging.

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12. The process of Claim 11, wherein said particles produced are white.

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13. The process of Claim 12, further including the step of dying said white particles another color.

14. The process of Claim 13, wherein said dying is performed by immersing said white particles in an aqueous solution of a metal oxide.

15. The process of Claim 14, wherein said metal oxide is selected from the group comprising ruthenium tetroxide and osmium tetroxide.

5 16. The process of Claim 9, wherein said second monomer is methacrylic acid.

10 17. The process of Claim 16, wherein said functional monomer is selected from the group consisting of acrylic acid, sodium styrene sulfonate, maleic acid, chlorostyrene and vinyl alcohol, producing dielectric particles with an acidic surface suitable for negative charging.

18. The process of Claim 17, wherein said particles produced are white.

15 19. The process of Claim 18, further including the step of dyeing said white particles another color.

20 20. The process of Claim 19, wherein said dyeing is performed by immersing said white particles in an aqueous solution of a metal oxide.

21. The process of Claim 20, wherein said metal oxide is selected from the group comprising ruthenium tetroxide and osmium tetroxide.

22. The process of Claim 3, wherein said step of allowing said first monomer to polymerize includes agitating and warming said third mixture.

23. The process of Claim 3, further including the step of separating said polymer particles from said third mixture by centrifuging and decanting the supernatant liquid.

24. Dielectric particles formed by the process of Claim 3.

25. Dielectric particles formed by the process of Claim 14.

26. Dielectric particles formed by the process of Claim 16.

27. Dielectric particles formed by the process of Claim 20.

28. A process for forming an electrophoretic fluid for use in an electrophoretic display, comprising:

- (a) providing a dielectric fluid;
- (b) preparing dielectric particles by a two stage polymerization process; and
- (c) dispersing said dielectric particles in said dielectric fluid.

29. The process of Claim 28, wherein said step of preparing includes admixing a first monomer and a crosslinker in a liquid dispersion medium forming a first mixture;

preparing a second mixture of an initiator and a stabilizer;

5 adding said first mixture to said second mixture to form a third mixture;

allowing said first monomer to polymerize within said third mixture to form polymer particles;

10 introducing a second monomer to said third mixture, said second monomer at least partially polymerizing and grafting upon said polymer particles; and

introducing a functional monomer to said third mixture when said second monomer is introduced.

15 30. The process of Claim 29, further including the steps of centrifuging said third mixture to separate said dielectric particles from said dispersion medium and decanting the supernatant said dispersion medium prior to said step of dispersing said particles in said dielectric fluid.

31. The process of Claim 30, further including the steps of resolving said dielectric particles after centrifuging said third mixture with a solvent miscible with said dispersion medium and with said dielectric fluid and further centrifuging said particles and decanting said solvent prior to dispersing said particles in said dielectric fluid.

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32. The process of Claim 31, further including the step of adding a charge control agent to said dielectric fluid after said step of dispersing in said dielectric fluid.

33. The process of Claim 32, wherein said charge control agent is an acidic surfactant and a positive charge control agent.

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34. The process of Claim 33, wherein said positive charge control agent is selected from the group consisting of polyisobutylene succinic anhydride, cupric naphthenate, zirconium octoate, zinc octoate, calcium octoate, polyvinyl alcohol, polyacrylic acid, polymethacrylic acid and polyvinyl chloride.

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35. The process of Claim 32, wherein said charge control agent is a basic surfactant and a negative charge control agent.

36. The process of Claim 35, wherein said negative charge control agent is selected from the group consisting of polyisobutylene succinimide, barium petronate, barium sulfonate, barium dinonylnaphthalensulfonate, metal oxides, polyvinyl pyridine, pyridine, lecithin, polyvinyl acetate, polyethelene oxide, polymethy methacrylate, polyacrylamide, polyesters and polyethers.

37. The process of Claim 32, further including adding a dye to said dielectric fluid when said charge control agent is added.

38. The process of Claim 37, wherein said dye is selected from the group consisting of Solvent Blue, Oil Blue A, Sudan Black B, and Sudan Red 7B.

39. The process of Claim 32, further including the step of adding a stabilizer to said dielectric fluid when said charge control agent is added.

40. The process of Claim 39, wherein said stabilizer is selected from the group consisting of homopolymers, copolymers and block copolymers.

41. The process of Claim 39, wherein said stabilizer is selected from

the group consisting of poly(12-hydroxystearic acid)-graft-poly(methyl methacrylate-methacrylic acid), polystyrene-co-poly(vinyl pyridine) and poly(vinyl alcohol)-co-polyethylene.

5 42. The process of Claim 28, wherein said dielectric fluid is a non-polar solvent.

10 43. The process of Claim 42 wherein said dielectric fluid is selected from the group consisting of tetrachloroethylene, carbon tetrachloride, pentane, octane, decane, cyclohexane, benzene, xylene, sec-butylbenzene and Isopars.

15 44. The process of Claim 31, wherein said solvent is selected from the group consisting of ethanol, propanol, butanol, acetone, tetrahydrofuran, ketones, ethers, and esters.

20 45. The process of Claim 34, wherein said first monomer is selected from the group consisting of styrene, methyl methacrylate, vinyl acetate, acrylate, ethyl vinylbenzene, vinylpyridine, and acrylonitrile, said crosslinker is selected from the group consisting of nonconjugated divinyl compounds, diacrylate compounds, triacrylate compounds, dimethacrylate compounds and trimethacrylate compounds, said initiator is selected from the group consisting of 2,2'-azobisisobutyronitrile, 4,4'-azobis(r-cyanopentanic acid), 2,2'-azobis(2-

5 methylbutyronitrile), benzoyl peroxide, lauroyl peroxide and octanoyl peroxide, said stabilizer is selected from the group consisting of poly(vinyl pyrrolidone), homopolymers, copolymers, graft polymers, block polymers, poly(acrylic acid), poly(vinyl alcohol), poly(methacrylic acid) and sorbitan stearic acid monoester and said second monomer is acrylamide.

10 46. The process of Claim 45, wherein said functional monomer is selected from the group consisting of vinyl acetate, methyl methacrylate, acrylonitrile, acrylamide and dimethylaminopropylmethacrylamide and produce dielectric particles with a basic surface suitable for positive charging.

15 47. The process of Claim 36, wherein said first monomer is selected from the group consisting of styrene, methyl methacrylate, vinyl acetate, acrylate, ethyl vinylbenzene, vinylpyridine, and acrylonitrile, said crosslinker is selected from the group consisting of nonconjugated divinyl compounds, diacrylate compounds, triacrylate compounds, dimethacrylate compounds and trimethacrylate compounds, said initiator is selected from the group consisting of 2,2'-azobisisobutyronitrile, 4,4'-azobis(r-cyanopentanic acid), 2,2'-azobis(2-methylbutyronitrile), benzoyl peroxide, lauroyl peroxide and octanoyl peroxide, said stabilizer is selected from the group consisting of poly(vinyl pyrrolidone), homopolymers, copolymers, graft polymers, block polymers, poly(acrylic acid), poly(vinyl alcohol), poly(methacrylic acid) and sorbitan stearic acid monoester

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and said second monomer is methacrylic acid.

- 5 48. The process of Claim 47, wherein said functional monomer is selected from the group consisting of acrylic acid, sodium styrene sulfonate, maleic acid, chlorostyrene and vinyl alcohol, producing dielectric particles with an acidic surface suitable for negative charging.

AMENDED CLAIMS

[received by the International Bureau on 20 July 1994 (20.07.1994);
original claims 1-48 replaced by amended claims 1-42 (10 pages)]

1. A process for forming dielectric particles, comprising:
admixing a first monomer and a crosslinker in a dispersion
medium;
5 adding an initiator and a stabilizer to said dispersion medium so
that said first monomer polymerizes to form said dielectric particles; and
introducing a second monomer and a functional monomer to said
dispersion medium so that said second monomer and said functional monomer
can at least partially polymerize and graft onto said dielectric particles to form
10 a thin functional polymer coating which controls the charging of said dielectric
particles.
2. The process of Claim 1, wherein said first monomer is a vinyl
monomer.
- 15 3. The process of Claim 1, wherein said first monomer is selected
from the group consisting of styrene, methyl methacrylate, vinyl acetate, acrylate,
ethyl vinylbenzene, vinylpyridine, and acrylonitrile.
- 20 4. The process of Claim 3, wherein said crosslinker is a compound
having a plurality of polymerizable double bonds.

5. The process of Claim 4, wherein said crosslinker is selected from the group consisting of nonconjugated divinyl compounds, diacrylate compounds, triacrylate compounds, dimethacrylate compounds and trimethacrylate compounds.

6. The process of Claim 5, wherein said initiator is selected from the group consisting of 2,2'-azobisisobutyronitrile, 4,4'-azobis(r-cyanopentanoic acid), 2,2'-azobis(2-methylbutyronitrile), benzoyl peroxide, lauroyl peroxide and octanoyl peroxide.

7. The process of Claim 6, wherein said stabilizer is selected from the group consisting of poly(vinyl pyrrolidone), homopolymers, copolymers, graft polymers, block polymers, poly(acrylic acid), poly(vinyl alcohol), poly(methacrylic acid) and sorbitan stearic acid monoester.

8. The process of Claim 7, wherein said second monomer is acrylamide.

9. The process of Claim 8, wherein said functional monomer is selected from the group consisting of vinyl acetate, methyl methacrylate, acrylonitrile, N-(iso-utoxymethyl) acrylamide and

dimethylaminopropylmethacrylamide and produce dielectric particles with a basic surface suitable for positive charging.

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10. The process of Claim 9, wherein said particles produced are white.

11. The process of Claim 10, further including the step of dying said white particles another color.

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12. The process of Claim 11, wherein said dying is performed by immersing said white particles in an aqueous solution of a metal oxide.

13. The process of Claim 12, wherein said metal oxide is selected from the group comprising ruthenium tetroxide and osmium tetroxide.

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14. The process of Claim 7, wherein said second monomer is methacrylic acid.

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15. The process of Claim 14, wherein said functional monomer is selected from the group consisting of acrylic acid, sodium styrene sulfonate, maleic acid, chlorostyrene and vinyl alcohol, producing dielectric particles with an acidic surface suitable for negative charging.

16. The process of Claim 15, wherein said particles produced are white.

5 17. The process of Claim 16, further including the step of dying said white particles another color.

18. The process of Claim 17, wherein said dying is performed by immersing said white particles in an aqueous solution of a metal oxide.

10 19. The process of Claim 18, wherein said metal oxide is selected from the group comprising ruthenium tetroxide and osmium tetroxide.

20. The process of Claim 1, wherein said step of allowing said first monomer to polymerize includes agitating and warming said dispersion medium.

15 21. The process of Claim 1, further including the step of separating said dielectric particles from said dispersion medium by centrifuging and decanting the supernatant liquid.

20 22. A process for forming an electrophoretic fluid for use in an electrophoretic display, comprising:

admixing a first monomer and a crosslinker in a dispersion

medium; said dispersion medium being selected from the group consisting of alcohols, ethers, halogenated hydrocarbons, ketones and esters;

adding an initiator and a stabilizer to said dispersion medium so that said first monomer polymerizes to form dielectric polymer particles;

5 introducing a second monomer to said dispersion medium, said second monomer at least partially polymerizing and grafting upon said dielectric polymer particles;

separating said dielectric polymer particles from said dispersion medium; and

10 dispersing said dielectric particles in a dielectric fluid.

23. The process of Claim 22, further including the step of introducing a functional monomer to said dispersion medium when said second monomer is introduced.

15 24. The process of Claim 23, wherein said step of separating said dielectric particles from said dispersion medium includes centrifuging said dispersion medium and decanting the supernatant of said dispersion medium prior to said step of dispersing said particles in said dielectric fluid.

20 25. The process of Claim 24, further including the steps of resolating said dielectric particles after centrifuging said dispersion medium with a solvent

miscible with said dispersion medium and with said dielectric fluid and further centrifuging said particles and decanting said solvent prior to dispersing said particles in said dielectric fluid.

5 26. The process of Claim 25, further including the step of adding a charge control agent to said dielectric fluid after said step of dispersing in said dielectric fluid.

10 27. The process of Claim 26, wherein said charge control agent comprises an acidic surfactant and a positive charge control agent.

15 28. The process of Claim 27, wherein said positive charge control agent is selected from the group consisting of polyisobutylene succinic anhydride, cupric naphthenate, zirconium octoate, zinc octoate, calcium octoate, polyvinyl alcohol, polyacrylic acid, polymethacrylic acid and polyvinyl chloride.

 29. The process of Claim 26, wherein said charge control agent comprises a basic surfactant and a negative charge control agent.

20 30. The process of Claim 29, wherein said negative charge control agent is selected from the group consisting of polyisobutylene succinimide, barium petronate, barium sulfonate, barium

dinonylnaphthalensulfonate, metal oxides, polyvinyl pyridine, pyridine, lecithin, polyvinyl acetate, polyethelene oxide, polymethy methacrylate, polyacrylamide, polyesters and polyethers.

5 31. The process of Claim 26, further including adding a dye to said dielectric fluid when said charge control agent is added.

10 32. The process of Claim 31, wherein said dye is selected from the group consisting of Solvent Blue, Oil Blue A, Sudan Black B, and Sudan Red 7B.

 33. The process of Claim 26, further including the step of adding a stabilizer to said dielectric fluid when said charge control agent is added.

15 34. The process of Claim 33, wherein said stabilizer is selected from the group consisting of homopolymers, copolymers and block copolymers.

20 35. The process of Claim 33, wherein said stabilizer is selected from the group consisting of poly(12-hydroxystearic acid)-graft-poly(methyl methacrylate-methacrylic acid), polystyrene-co-poly(vinyl pyridine) and poly(vinyl alcohol)-co-polyethylene.

36. The process of Claim 22, wherein said dielectric fluid is a non-polar solvent.

5 37. The process of Claim 36 wherein said dielectric fluid is selected from the group consisting of tetrachloroethylene, carbon tetrachloride, pentane, octane, decane, cyclohexane, benzene, xylene, sec-butylbenzene and Isopars.

10 38. The process of Claim 25, wherein said solvent is selected from the group consisting of ethanol, propanol, butanol, acetone, tetrahydrofuran, ketones, ethers, and esters.

15 39. The process of Claim 28, wherein said first monomer is selected from the group consisting of styrene, methyl methacrylate, vinyl acetate, acrylate, ethyl vinylbenzene, vinylpyridine, and acrylonitrile, said crosslinker is selected from the group consisting of nonconjugated divinyl compounds, diacrylate compounds, triacrylate compounds, dimethacrylate compounds and trimethacrylate compounds, said initiator is selected from the group consisting of 2,2'-azobisisobutyronitrile, 4,4'-azobis(r-cyanopentanic acid), 2,2'-azobis(2-methylbutyronitrile), benzoyl peroxide, lauroyl peroxide and octanoyl peroxide, 20 said stabilizer is selected from the group consisting of poly(vinyl pyrrolidone), homopolymers, copolymers, graft polymers, block polymers, poly(acrylic acid),

poly(vinyl alcohol), poly(methacrylic acid) and sorbitan stearic acid monoester and said second monomer is acrylamide.

5 40. The process of Claim 39, wherein said functional monomer is selected from the group consisting of vinyl acetate, methyl methacrylate, acrylonitrile, acrylamide and dimethylaminopropylmethacrylamide and produce dielectric particles with a basic surface suitable for positive charging.

10 41. The process of Claim 30, wherein said first monomer is selected from the group consisting of styrene, methyl methacrylate, vinyl acetate, acrylate, ethyl vinylbenzene, vinylpyridine, and acrylonitrile, said crosslinker is selected from the group consisting of nonconjugated divinyl compounds, diacrylate compounds, triacrylate compounds, dimethacrylate compounds and trimethacrylate compounds, said initiator is selected from the group consisting of
15 2,2'-azobisisobutyronitrile, 4,4'-azobis(r-cyanopentanic acid), 2,2'-azobis(2-methylbutyronitrile), benzoyl peroxide, lauroyl peroxide and octanoyl peroxide, said stabilizer is selected from the group consisting of poly(vinyl pyrrolidone), homopolymers, copolymers, graft polymers, block polymers, poly(acrylic acid), poly(vinyl alcohol), poly(methacrylic acid) and sorbitan stearic acid monoester
20 and said second monomer is methacrylic acid.

5 42. The process of Claim 41, wherein said functional monomer is selected from the group consisting of acrylic acid, sodium styrene sulfonate, maleic acid, chlorostyrene and vinyl alcohol, producing dielectric particles with an acidic surface suitable for negative charging.

FIG. 1

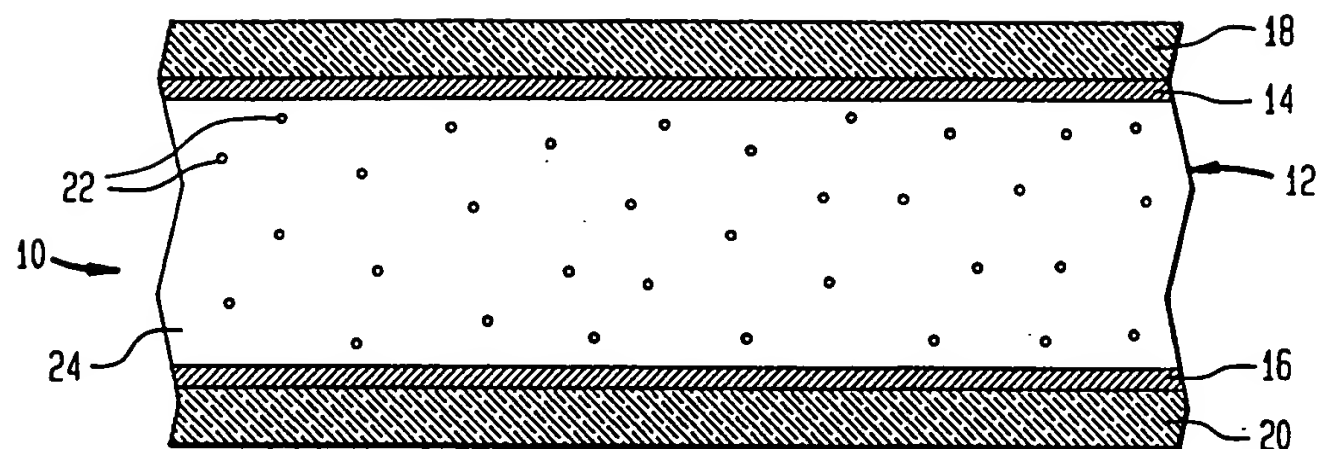
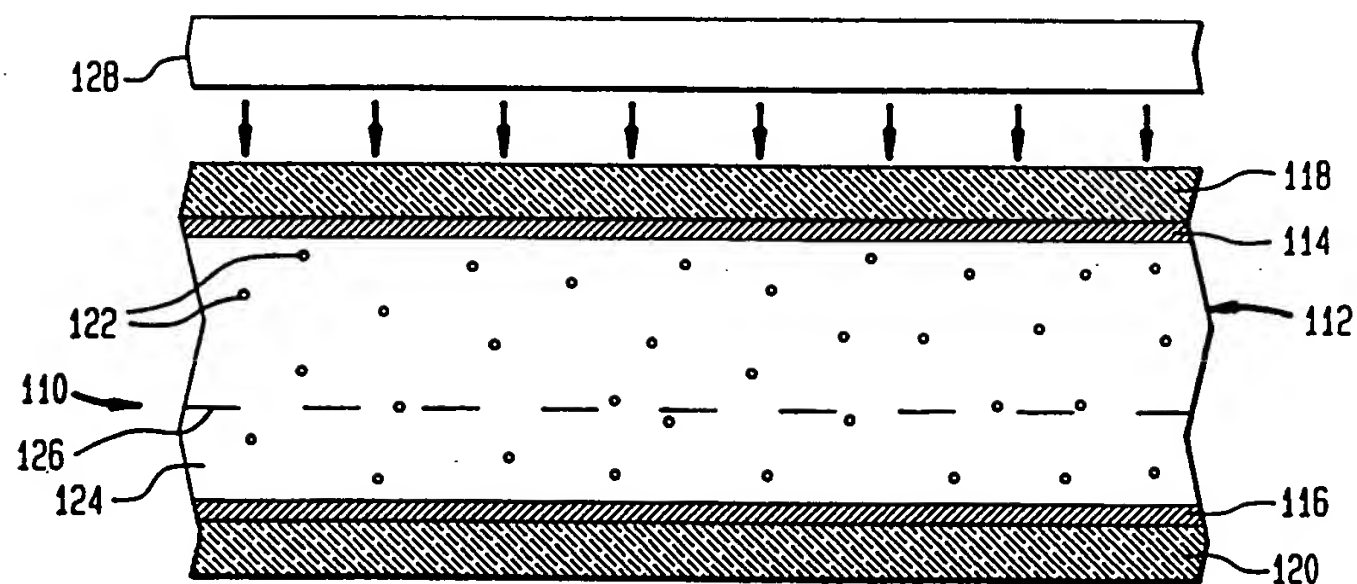


FIG. 2



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/03813

A. CLASSIFICATION OF SUBJECT MATTER

IPC(S) : C10M 169/04; C09K 3/00

US CL : 252/73, 77, 79, 572; 313/483; 204/299R

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 252/73, 77, 79, 572; 313/483; 204/299R

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
CAS styrene, methacrylic, crosslink, stabilize, electrophoretic

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 4,992,192 (AHMED) 12 FEBRUARY 1991 (Note column 4, line 20 - column 5, line 19).	1-48
Y	Journal of Polymer Science: Part A: Polymer Chemistry Edition, Vol. 24, 1986, Tseng et al, "Uniform Polymer Particles by Dispersion Polymerization in Alcohol", pages 2995-3007.	1-27

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	* T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
* A* document defining the general state of the art which is not considered to be of particular relevance	* X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
* E* earlier document published on or after the international filing date	* Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
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* I* document referring to an oral disclosure, use, exhibition or other means	
* P* document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

03 JUNE 1994

Date of mailing of the international search report

JUN 21 1994

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer
CHRISTINE SKANE

Telephone No. (703) 308-2526

in the composite matrix in response to the energy supplied by the field in their entirety arrive in the electrical or non-conducting state.

10. Electric field generator/modulator (EFGM) for patterning and generating electrical conducting or semiconducting structures in two or three dimensions in a composite matrix, wherein the matrix comprises one or more materials respectively provided in one or more spatially separate and homogenous material structures, wherein the materials in response to the supplied energy can undergo specific and/or chemical changes of state which cause transition from an electrical non-conducting state to an electrical conducting or semiconducting state and vice versa, or a change in the conduction mode of the material, wherein each material structure is made in the form of a thin layer, and wherein the electric field generator/modulator (20) is characterized in that it comprises a first electrode means (E1) with a plurality of parallel strip electrodes (21) provided in a plane, a second electrode means (E2) with a plurality of parallel strip electrodes (22) provided at a distance from the first electrode means (E1) and superpositioned thereto in a second plane parallel with the first plane such that the electrodes (21, 22) mutually are substantially orthogonally oriented in a matrix-like arrangement, that the electrode means (E1;E2) over cross-connection devices (24;25) are connected with a controllable power supply (23), the electrical field generator/modulator (20) in the space between the electrode means (E1, E2) being adapted for receiving a thin-film material in the form of a discrete component or a continuous tape which without touching the electrode means continuously or intermittently is fed through the space with simultaneous positioning and alignment spaced apart from and between the electrode means (E1, E2) in a plane substantially parallel thereto, whereby the electrical conducting or semiconducting structures can be generated according to a determined protocol and by means of point, line or area potentials are created between selected electrodes (21, 22) in the electrode means (E1;E2) when the former over the cross-connection devices (24;25) are supplied with electric power.

11. Electric field generator/modulator according to claim 10, characterized in that the electrodes (21;22) in each electrode device (E1;E2) is provided on or in surfaces of respective substrates facing each other.

12. Electric field generator/modulator according to claim 11, characterized in that the strip electrodes (21, 22) are made as a part of the substrates and form conducting structures in the substrate material.
13. Electric field generator/modulator according to claim 10, characterized in that the distance between the electrode means (E1, E2) is controllable depending on the thickness of the thin-film material.
14. Electric field generator/modulator according to claim 10, characterized in that the electrodes (21;22) in each electrode means (E1; E2) are provided with a mutual distance between 0.1 μm and 1.0 μm .
15. Electric field generator/modulator according to claim 10, characterized in that the electrodes (21;22) in each electrode means (E1;E2) are formed with substantially constant width of 0.1 μm to 1.0 μm .

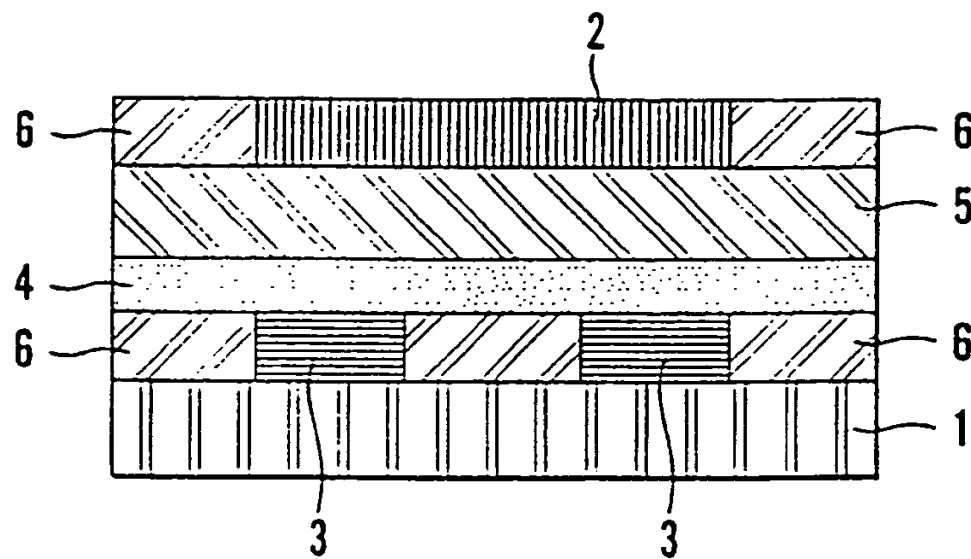


Fig. 1 (PRIOR ART)

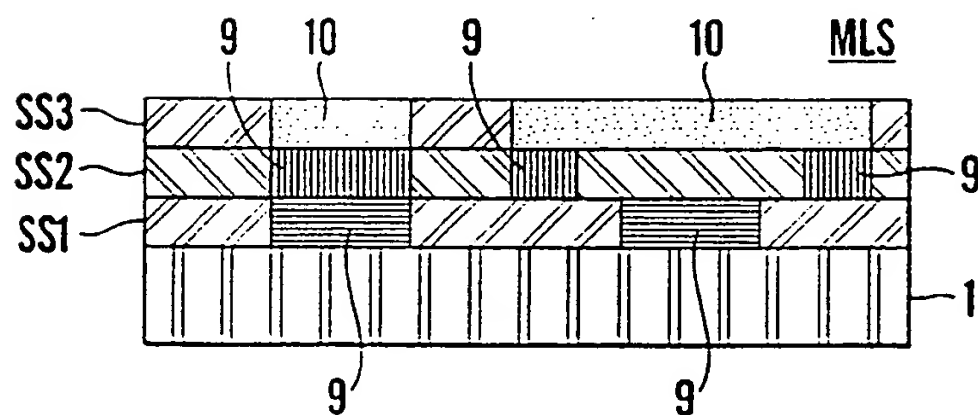


Fig. 4

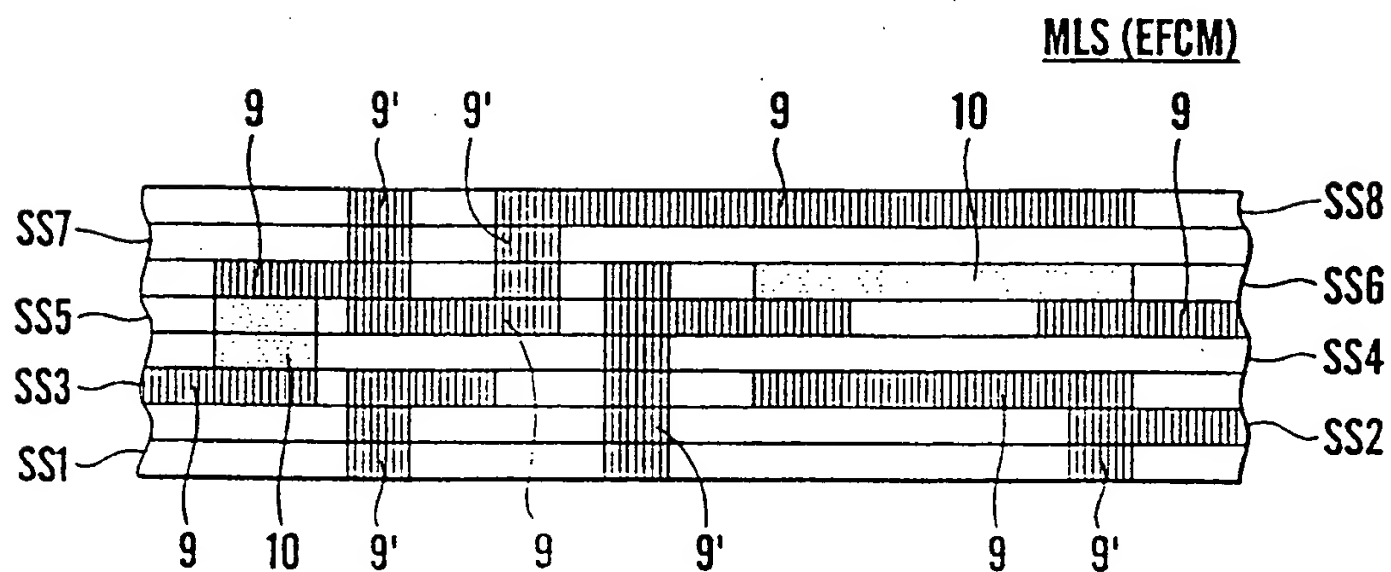


Fig. 5

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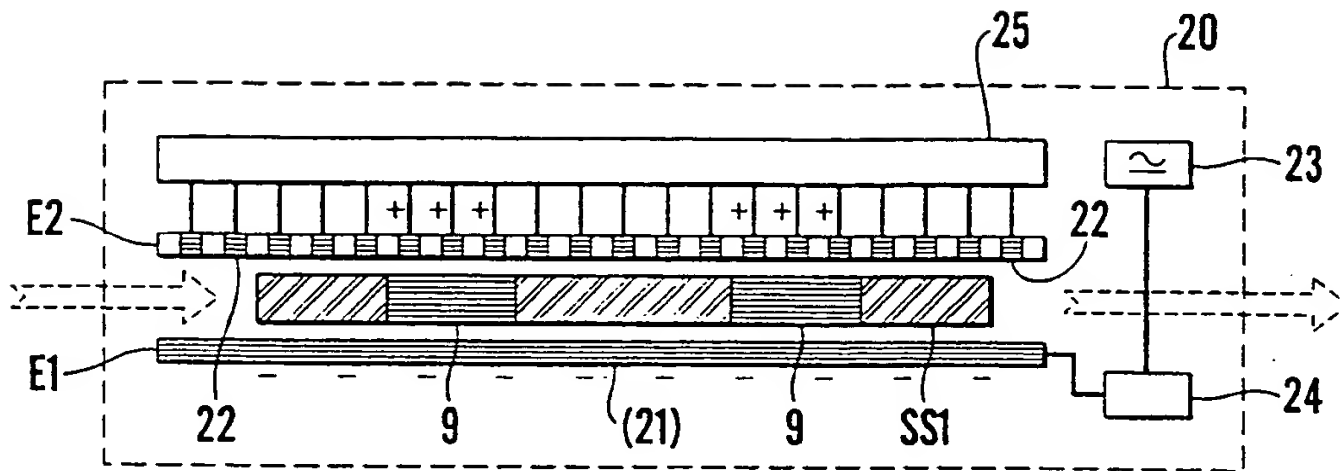


Fig. 2a

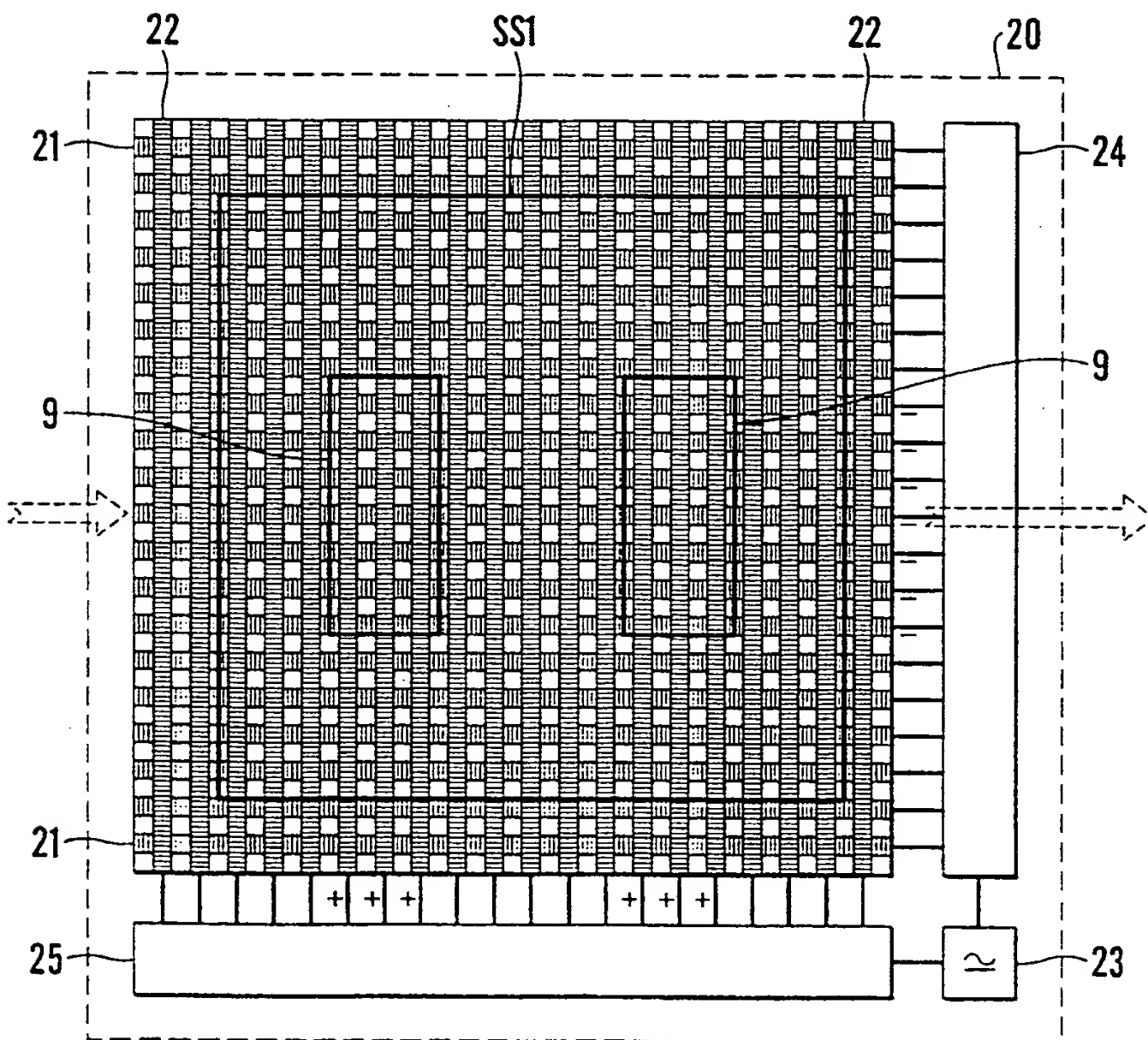


Fig. 2b

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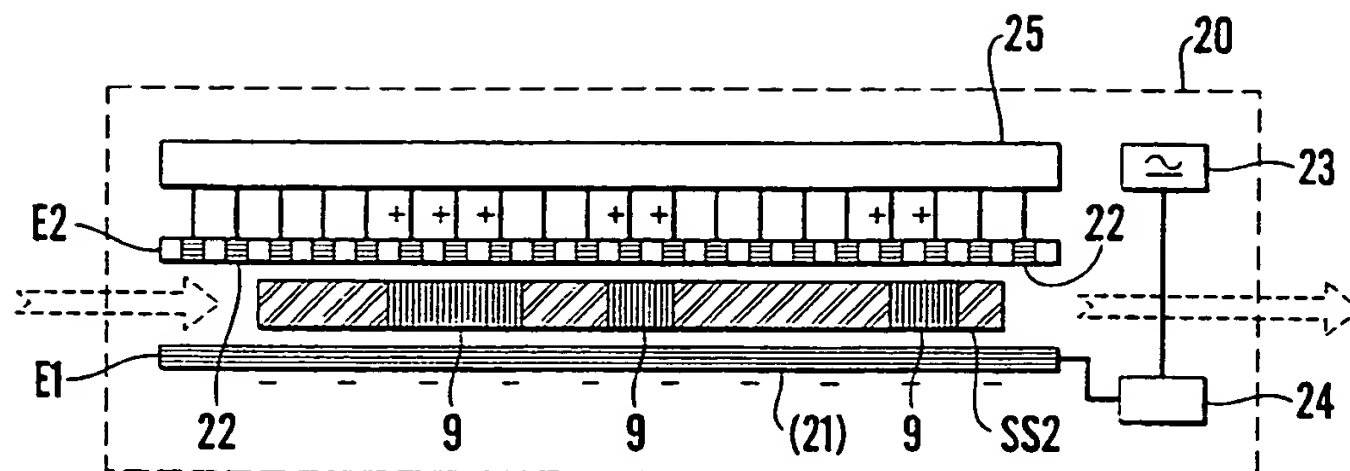


Fig. 2c

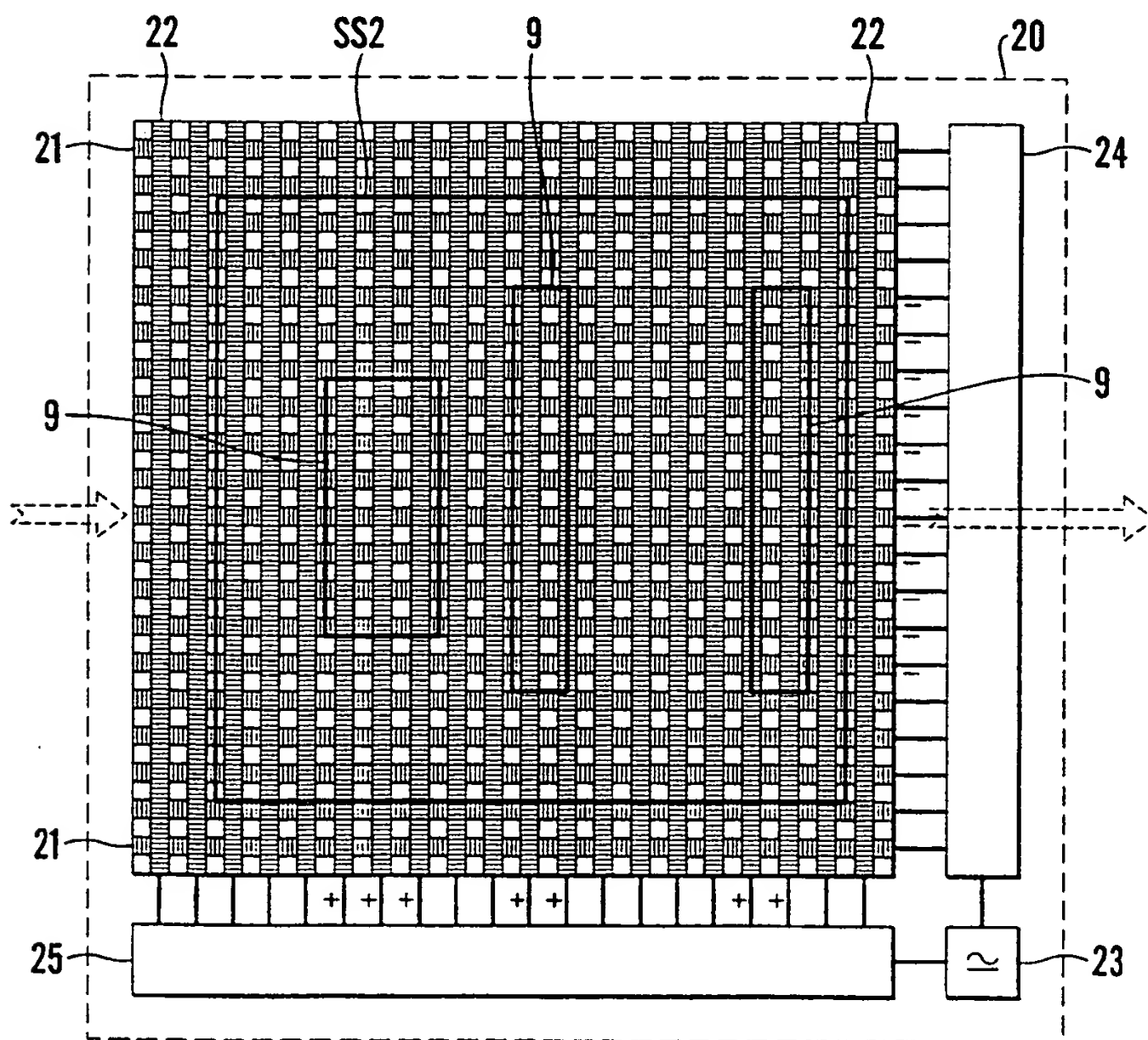


Fig. 2d

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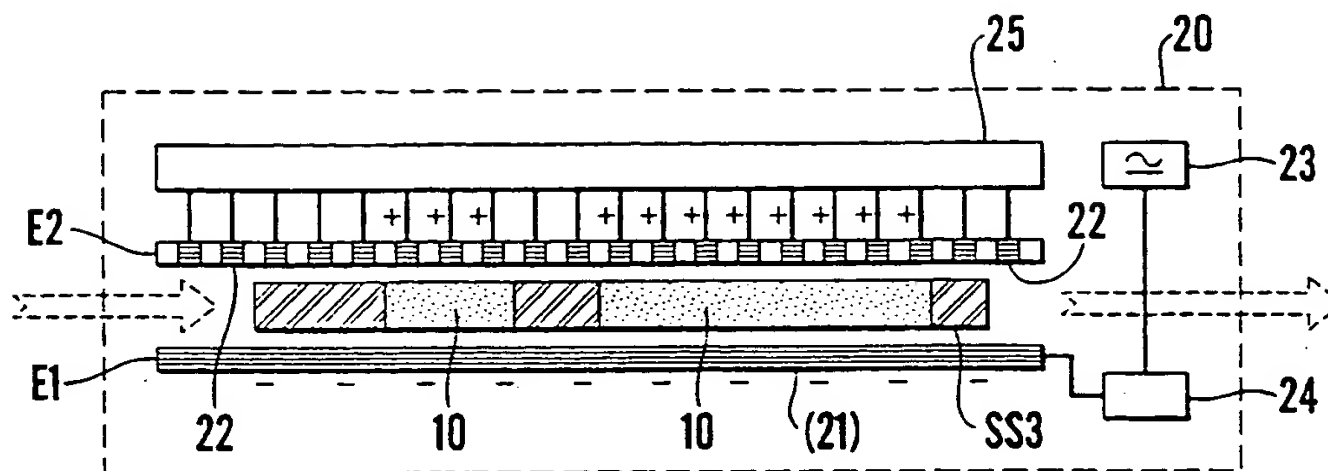


Fig. 2e

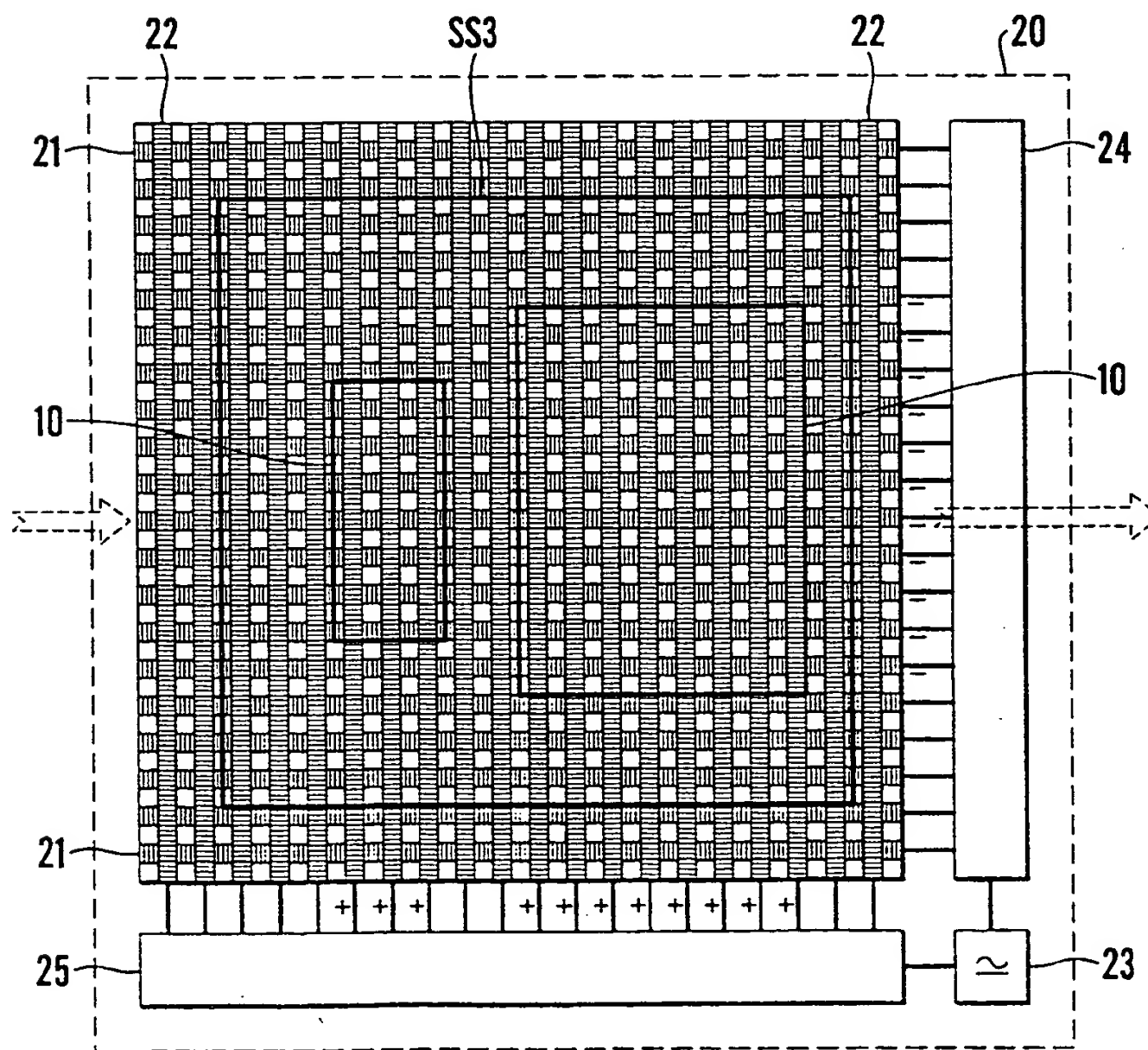


Fig. 2f

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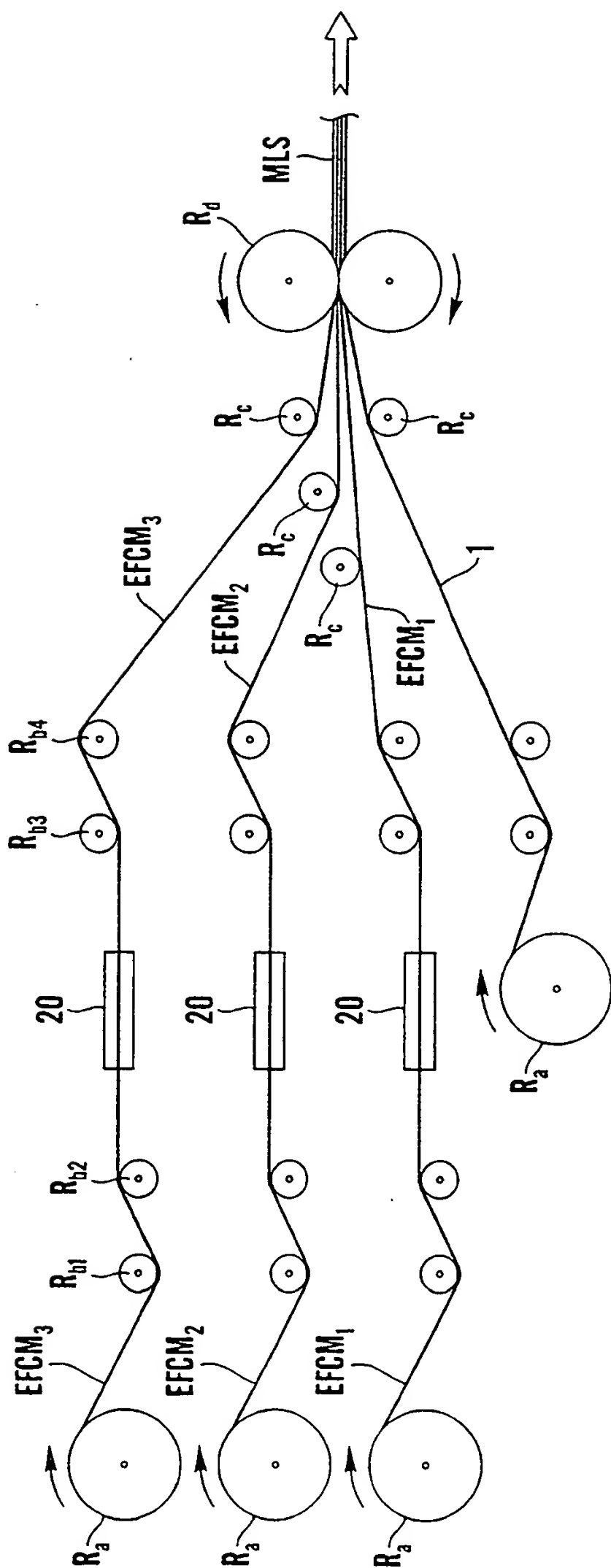







Fig.3

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LEGEND TO FIGS.

-  ISOLATING
 SEMICONDUCTING
 p-TYPE SEMICONDUCTING
 n-TYPE SEMICONDUCTING
 CONDUCTING, ELECTRODES

 HORIZONTAL AND VERTICAL CONDUCTING PATH (Δ , UP; ∇ , DOWN)

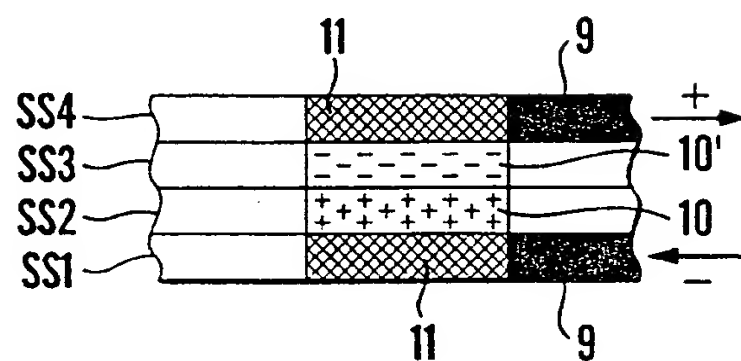
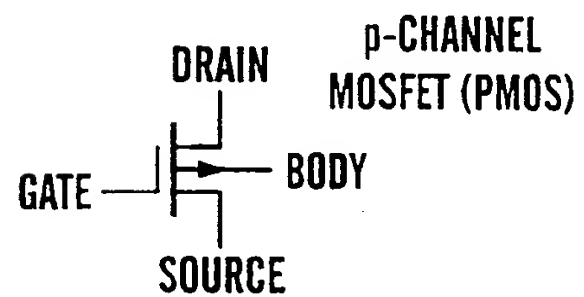
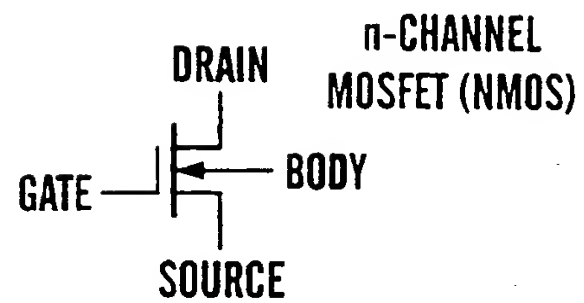


Fig.6

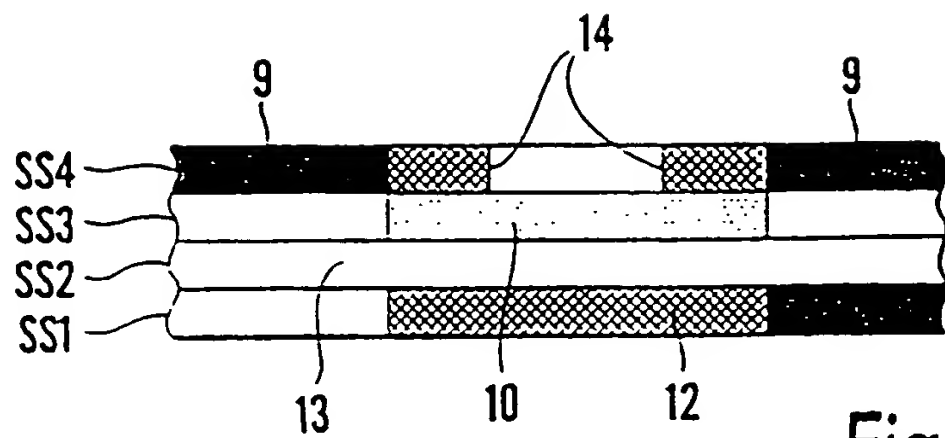


Fig. 7

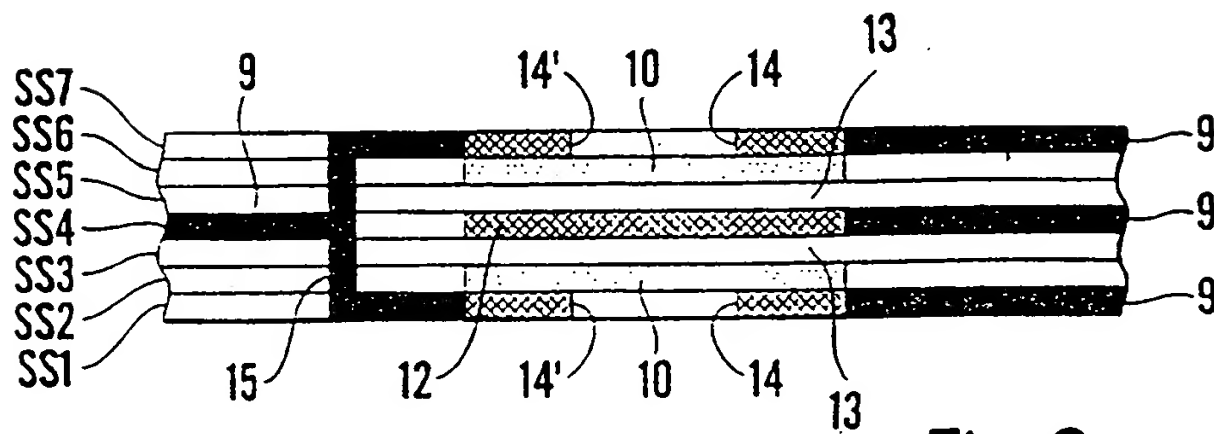


Fig. 8

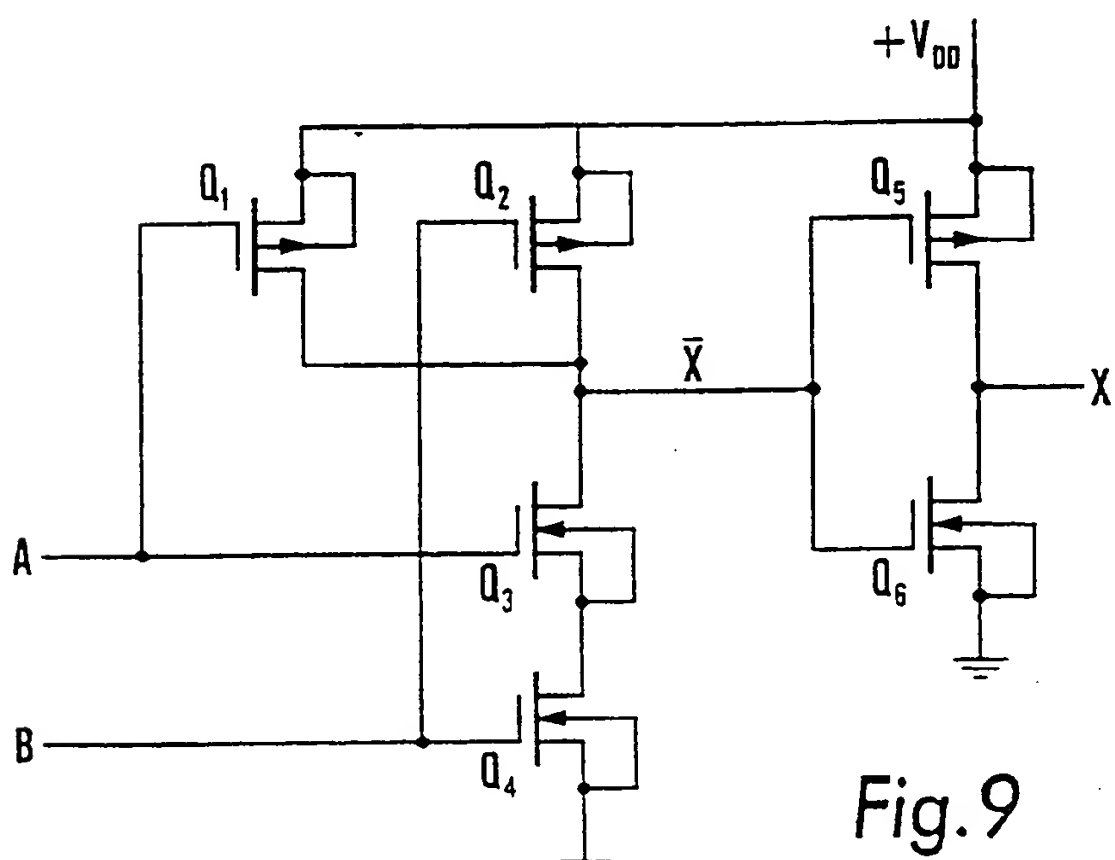


Fig. 9

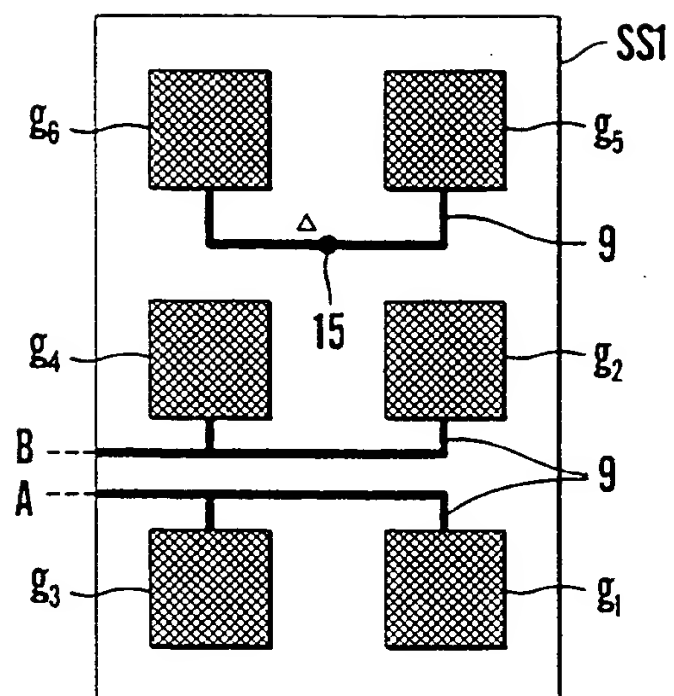


Fig. 10a

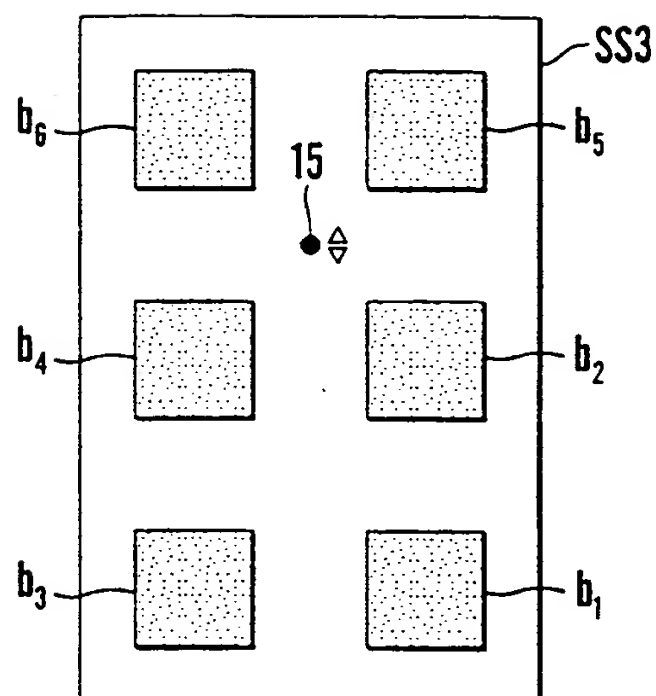


Fig. 10b

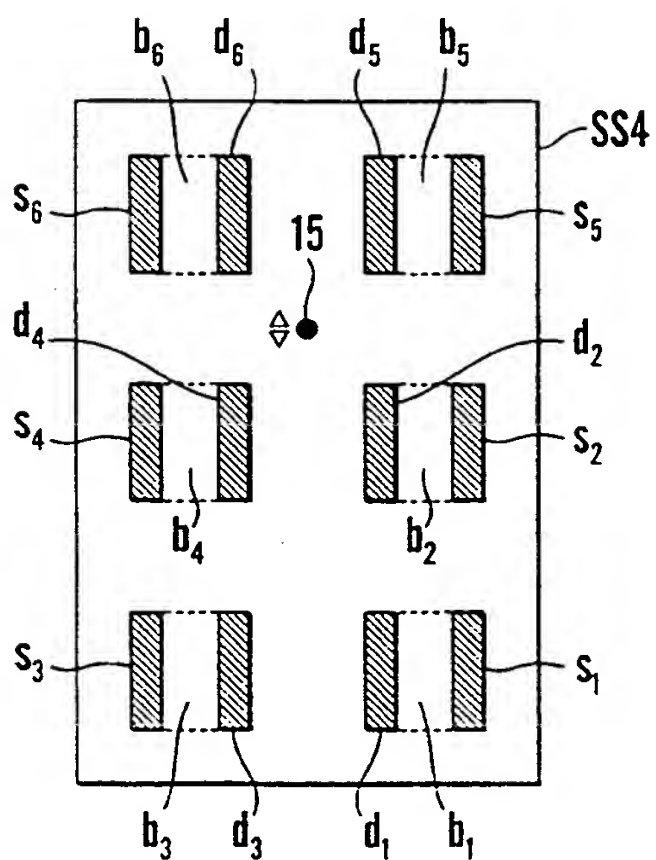


Fig. 10c

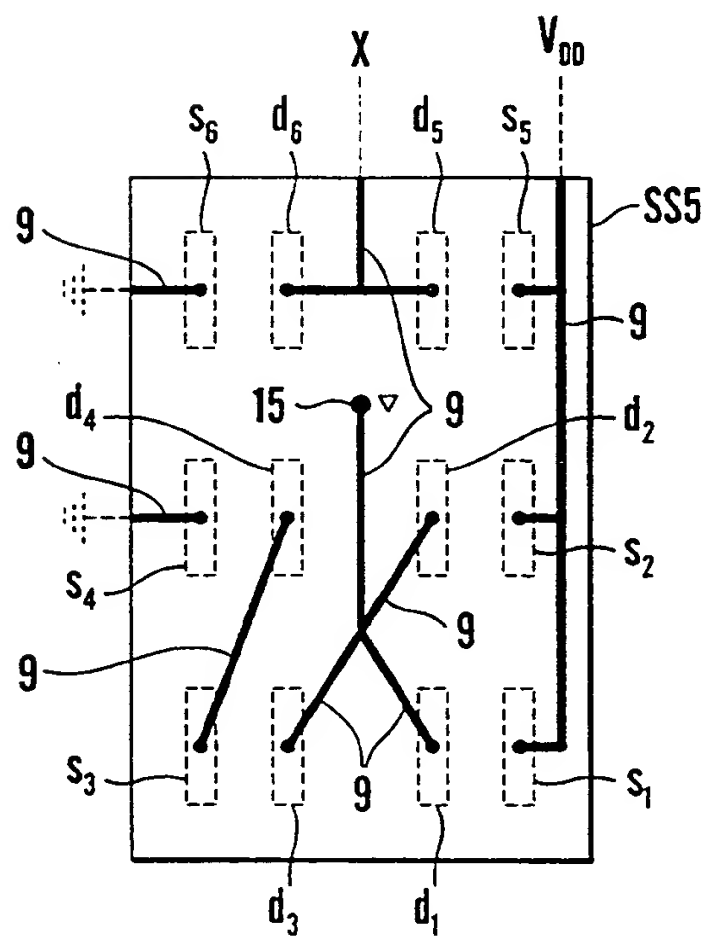


Fig. 10d

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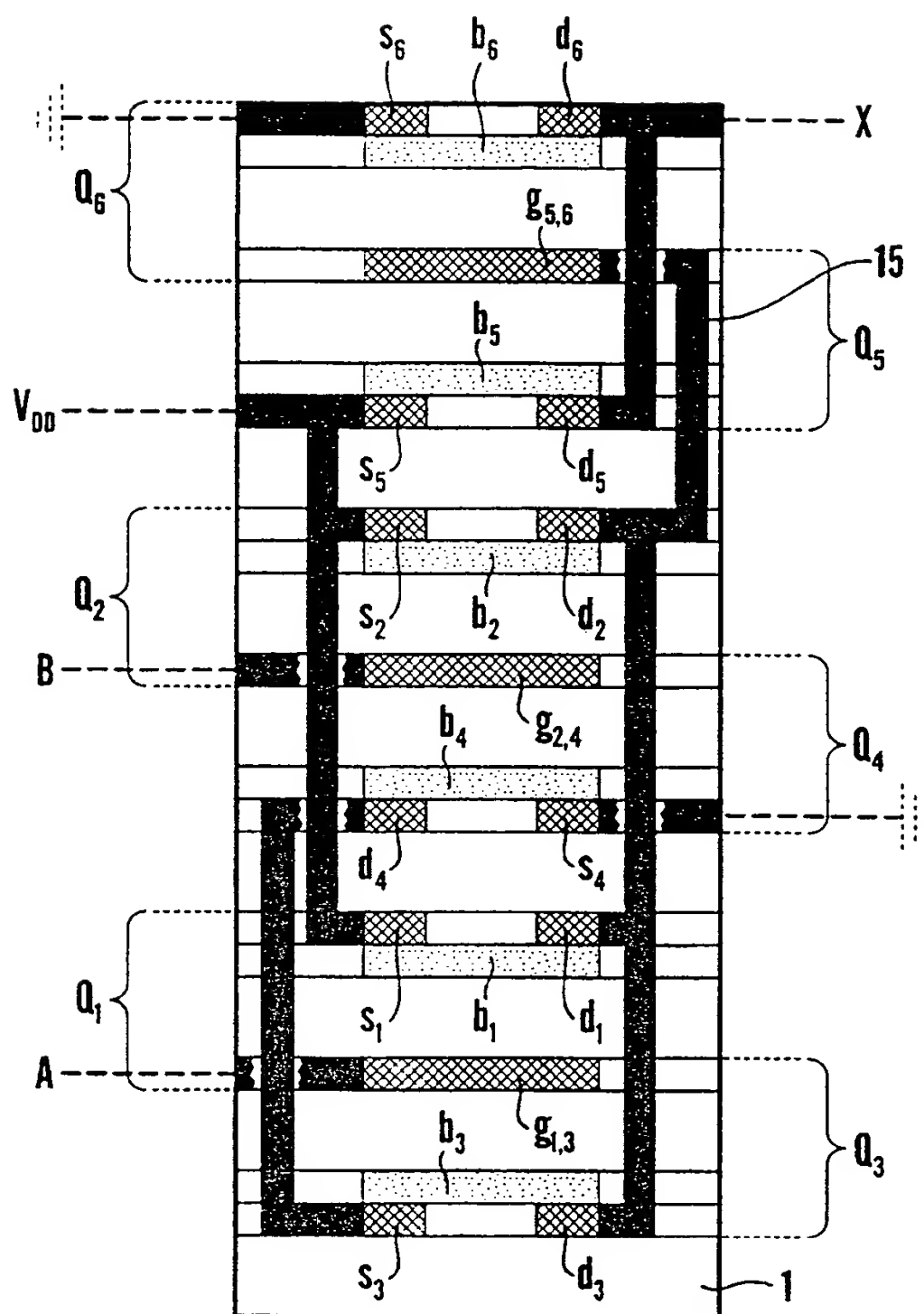


Fig. 12

INTERNATIONAL SEARCH REPORT

International application No. -

PCT/NO 99/00022

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: H01L 21/64, H01L 51/80

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: H01L, H05K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5572409 A (RICHARD J. NATHAN ET AL), 5 November 1996 (05.11.96), column 4, line 39 - line 60; column 5, line 17 - line 30	1,3-9
A	--	2,10-15
X	US 5537108 A (RICHARD J. NATHAN ET AL), 16 July 1996 (16.07.96), column 4, line 37 - line 58; column 5, line 15 - line 28	1,3-9
A	--	2,10-15

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:

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"P" document published prior to the international filing date but later than the priority date claimed

"I" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

23 July 1999

Date of mailing of the international search report

28-07-1999

Name and mailing address of the ISA/

Swedish Patent Office

Box 5055, S-102 42 STOCKHOLM

Facsimile No. +46 8 666 02 86

Authorized officer

Bengt Romedahl/cs

Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT

International application No.

PCT/NO 99/00022

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	US 5808351 A (RICHARD J. NATHAN ET AL), 15 Sept 1998 (15.09.98), column 4, line 37 - line 57; column 5, line 14 - line 27	1,3-9
A	--	2,10-15
A	US 5686341 A (BRUCE BOYD ROESNER), 11 November 1997 (11.11.97), abstract	1-15
A	--	1-15
	WO 9509438 A1 (KOPIN CORPORATION), 6 April 1995 (06.04.95), abstract	
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INTERNATIONAL SEARCH REPORT

Information on patent family members

01/07/99

International application No.

PCT/NO 99/00022

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5572409 A	05/11/96	US 5537108 A US 5726482 A US 5808351 A US 5813881 A US 5834824 A	16/07/96 10/03/98 15/09/98 29/09/98 10/11/98
US 5537108 A	16/07/96	US 5834824 A US 5572409 A US 5726482 A US 5808351 A US 5813881 A	10/11/98 05/11/96 10/03/98 15/09/98 29/09/98
US 5808351 A	15/09/98	US 5834824 A US 5537108 A US 5572409 A US 5726482 A US 5813881 A	10/11/98 16/07/96 05/11/96 10/03/98 29/09/98
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